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Effectiveness on color and COD of textile wastewater removing by biological material obtained from *Cassia fistula* seed

Hiệu quả xử lý màu và COD của nước thải dệt nhuộm bằng vật liệu sinh học được chiết xuất từ hạt Muồng Hoàng Yến

Research article

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Nowadays, natural polymeric materials extracted from plants are the new alternatives for synthetic chemicals in water and wastewater treatment. The aim of this study is to evaluate the ability of Cassia *fistula* seed gum (CFG) as a coagulant aid with PAC in the treatment of textile wastewater. Jartest experiments were carried out to identify the optimal parameters of coagulation-flocculation for removing color and COD in synthesis wastewater containing Methyl blue and RB21 dyes, including pH, settling time, PAC dose, the optimal CFG dosage in comparing with the cationic polymer. After that, actual textile wastewater was treated by using PAC, PAC plus cationic polymer, and PAC plus CFG for evaluating the role of CFG. CFG supplementation has assisted the process effects at nearly 98% color, 85% COD for RB21 and 90% color, 70% COD for MB at the best dose of CFG 0.15 mL and 0.1 mL, respectively. The optimized parameters for the coagulation of real textile wastewater using PAC were pH = 6 and dose = 0.6 mL can removal 66% of color. By adding CFG to PAC, the efficient of treatment was increased about 70% even at the lower dosage of PAC and CFG (0.5 mL for each reagent). The yield of combining PAC and polymer was a little bit lower than PAC and CFG, for instant 68% color was decreased at the same condition. These achievements demonstrated a workable substitute of natural products such as Cassis *fistula* seed gum for synthetic chemical products in coagulation-flocculation process.

Hiện nay các loại vật liệu sinh học chiết xuất từ thực vật đang được nghiên cứu ứng dụng trong xử lý nước và nước thải thay cho các chất hóa học. Mục tiêu của nghiên cứu này là đánh giá hiệu quả của việc sử dụng gum được chiết xuất từ hạt cây Muồng Hoàng Yến (MHY) làm chất trợ keo tụ trong xử lý nước thải dệt nhuộm. Thí nghiệm Jartest được tiến hành nhằm xác định các điều kiện tối ưu cho quá trình xử lý nước thải tổng hợp chứa thuốc nhuộm Methyle Blue (MB) và RB21 bao gồm pH, thời gian lắng, liều PAC, liều gum MHY và liều polymer. Sau đó tiến hành xử lý nước thải thật với các điều kiện thích hợp đã xác định nhằm đánh giá vai trò của gum MHY. Gum MHY làm tăng hiệu quả của quá trình xử lý, đạt gần 98% đối với độ màu, 85% COD đối với RB21, 90% độ màu và 70% COD đối với MB với liều lượng tương ứng là 0,15 mL và 0,1 mL. Các thông số tối ưu cho quá trình xử lý trên mẫu nước thải thật là pH = 6, liều PAC = 0.6 mL có thể làm giảm 66% độ màu. Bổ sung gum MHY làm chất trợ keo tụ giúp gia tăng hiệu quả xử lý màu lên 70% dù với liều lượng rất thấp là 0,5 mL. Hiệu suất xử lý khi sử dụng kết hợp PAC và polymer thấp hơn trong trường hợp sử dụng PAC và gum MHY, cụ thể khoảng 68% độ màu được xử lý ở cùng một điều kiện. Những kết quả này cho thấy tiềm năng của việc sử dụng các vật liệu gum tự nhiên nhằm thay thế cho các hợp chất hóa học trong các quá trình keo tụ tạo bông để xử lý nước thải.

Keywords: COD removal, textile wastewater, Cassia *fistula* seed gum, coagulation, flocculation

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1. Introduction

The wastewater generated by the textile industry has become the most pollution source among all industrial sectors due to the high volume discharge, complex chemical composition, and high concentration of pollutants (Verma et al., 2012). The textile processing often generates about 200-350 m^3 of wastewater per ton of product. In Vietnam, textile wastewater has been reported as a high potential pollution source due to this industrial sector has developed rapidly, therefore increased the requirement of treating and controlling this wastewater. The main sources of wastewater generated by the textile wet-processing are the washing or scouring and bleaching of natural that response for 10% of dyes lost. Most of the wastewater produced by the textile industry is particularly associated with reactive azo dyes that are used for dyeing cellulose fibres. The effluent containing residual dyes, by-product, auxiliary chemical resulting in the high color and other pollutants (Hanif et al., 2007, 2008, Verma et al., 2012, Perng et al., 2015).

A number of methods applied in removing of color and COD of textile wastewater, including the combination of the chemical-physical treatment with a biological step, as well as some new emerging techniques like sonochemical or advanced oxidation. By comparing to other technologies, coagulation_flocculation is an effective method because of the economically feasible and high color removal capacity (Bratby, 2007, Verma et al., 2012). The most applicable technology is coagulation_flocclulation by chemical agents (PAC, Ferrous sulphate, Alum, Magnesium chloride) with the aid of chemical polymer in both laboratory research and in actual treatment (Perng et al., 2014, 2015).

Chemical coagulation and flocculation involves the addition of materials to stabilize the suspended particles (change the physical state of dissolved and suspended solids), allowing particle collision and growth of floc then removal them by sedimentation. In water treatment, coagulation is used as pretreatment for removal of suspended or colloidal materials that do not settle out on standing or may settle by taking a very long time coagulation (Metcalf & Eddy, 2007, Verma et al, 2012,). However, the major limitation of this process is the generation of sludge and ineffective decolourisation of some soluble dyes.

To improve the efficiency of coagulation process, a number of high molecular weight compounds such as synthetic polymers or natural origin may be recommended. These polymers can function as coagulant itself or in the form of coagulant aids/bioflocculants, depending upon the wastewater and polymer characteristics (Metcalf & Eddy (2007).

Nevertheless, chemical coagulants generally involve in less biodegradability and toxicity. For example, acrylamide is very much toxic and gives severe neurotoxic effects (Bratby, 2007). Toxicity effect due to cationic polymers to the plants has been established long back (Gao et al., 2001, 2007). The major advantage of natural polymer is its non-toxicity to the environment and biodegradability. Therefore, the effluent after natural polymer treatment can be treated by biological means and will not pose any harm to the biological organisms. The sludge generated by the natural polymers can further be treated biologically or can be disposed of safely as soil conditioners because of their non-toxicity (Yin, 2010, Subramonian, 2014). Hence, there is an urgent need to establish the use of natural low-cost polymers for textile wastewater treatment.

Natural polymeric materials extracted from plants are the new alternatives for synthetic polymers in water and wastewater treatment as they enhance the treatment's efficiency besides of prominent characteristics, such as highly biodegradable, non-toxic, non-corrosive, less sludge voluminous and pH independent. Nowadays, natural coagulants such as Guar gum, seed extract gum from Strychnos potato rum, Moringa Olifeira, and Cassia fistula, and others extraction from an animal (Chitosan) or micro-organism (Xanthan gum) have been used as a flocculation aid with the chemical coagulants. Cassia *obtusifolia* seed gum has the potential to treat 87% TSS and 55% COD comparable to those obtained in treatments using alum. In addition, the most prominent operating factors in the coagulation–flocculation process using the natural coagulant were the seed gum dosage, initial pH of wastewater and settling time (Damayanti, 2011, Shak & Wu, 2014, 2015; Choy et al., 2014).

Cassia *fistula* is recently planted in many areas in Vietnam since it has the beautiful flowers. Cassia fistula is commonly known as Amaltas in Pakistan, as Indian Labrum in India, as Canafistula in Brazil and as Golden Shower. It belongs to family Fabaceae, genus Cassia, species *Fistula*. Cassia *fistula* presents a high content of ionizable groups such as carboxyl, carbonyl, alcoholic and amino groups (Hanif et al. ,2007, 2008, Shak et al., 2015). Research of Mazhar Abbas (2007) examined the effect of various pretreatments on the capacities of Cassia fistula biomasses to remove Cr (III) and Cr (VI) and to study the effect of different conditions such pH, initial metal concentration, dosage, particle size and time require for the establishment of equilibrium. CFG was used in combination with PAC for decolorization of reactive dyeing wastewater and shown the decolorization efficiencies of both dyes reached over high values at 40 % volume fraction of gum A study in which Cassia *fistula* gum was used as an aid with PAC achieved the decolorization efficiencies of dyes containing reactive dyes Blue 19 (RB19) and Black 5 (RB5) reached over 92.6 % (RB19) and 94.2 % (RB5) at 40 % volume fraction of gum (Perng and Bui, 2014, 1015).

The performance of the coagulation_flocculation process has affected by selection of coagulant, flocculant aids, optimization of process parameters such as pH, the dosage of coagulant/flocculant aids, mixing time, and settling time (Hanif et al., 2007 Mazhar Abbas, 2007; Shak, 2014; Perng, 2014). A coagulation_flocculation system for textile wastewater treating should be designed based on the achieved optimum conditions.

This article is an attempt to evaluate the effectiveness of Cassia *fistula* seed gum as the coagulant aid with chemical coagulant for textile's wastewater treatment in the batch mode.

2. Materials and Methods

2.1. Materials

- The main coagulant used in this study is PAC (Polyaluminum chloride) has the density of 0.3 g/cm^3 and containing 30% Al_2O_3.

- Cassia *fistula* seed gum (CFG) was produced via the process that illustrated in figure 1. The seeds of the Cassia *fistula* tree were collected in Thu Dau Mot city, Binh Duong province, Vietnam when they became ripe and were dried under the sun. The dried seeds then were ground into powder in a blender before the extraction step. The polysaccharide from CF seed was extracted by Soxhlet system with quantity ratio 1:1 of n-hexane solution: powder for 24 hours. The fibrous mass was made by ethanol precipitation for 48 hours and then was dissolved in distilled water and precipitated again to purify the gum. In the last step, the gum was dried at 50^{0}C for 2 hours to obtain the complete CFG.

- The anionic polymer was used to assist the flocculation of the colloids after coagulation.

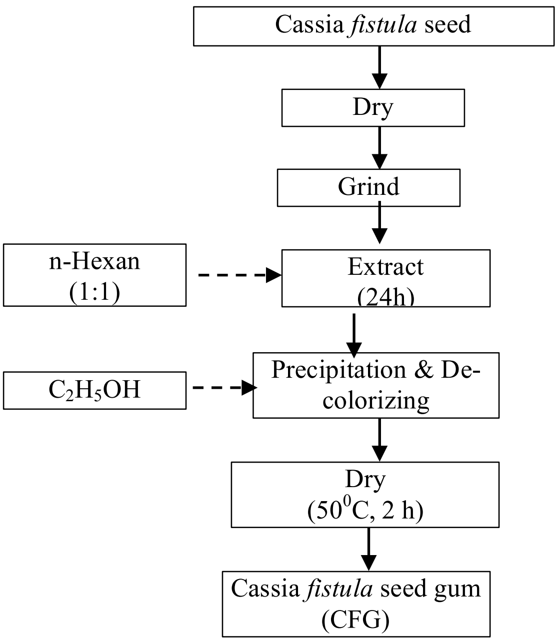

Figure 1. Cassia *fistula* seed gum producing process.

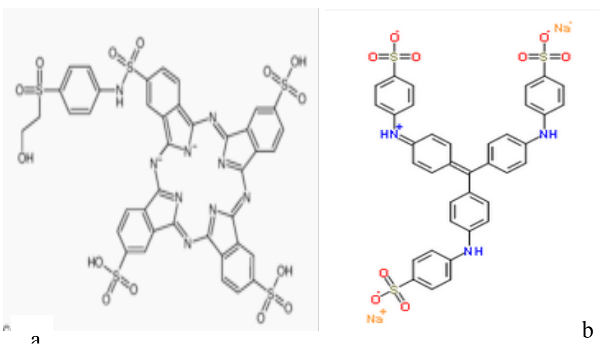

Figure 2. Chemical structure of RB21 (a) and MB (b)

- The synthetic wastewater was prepared by adding two familiar dyes in textile industry including RB21 – Reactive Blue 21 and Methyl Blue to determine the optimal conditions for the coagulation_flocculation process. The chemical structure of these materials as shown in figure 2.

- The actual textile wastewater was obtained at the Phuoc Long wastewater treatment plant for studying the effect of PAC coagulant in the aid of polymer and CFG.

2.2. Methods

In order to evaluate the feasibility of CFG as a coagulant with PAC for the treatment of textile wastewater at the pilot scale, it is necessary to conduct the batch experiments to determine the optimal conditions. Figure 3 illustrated the methodology of this study.

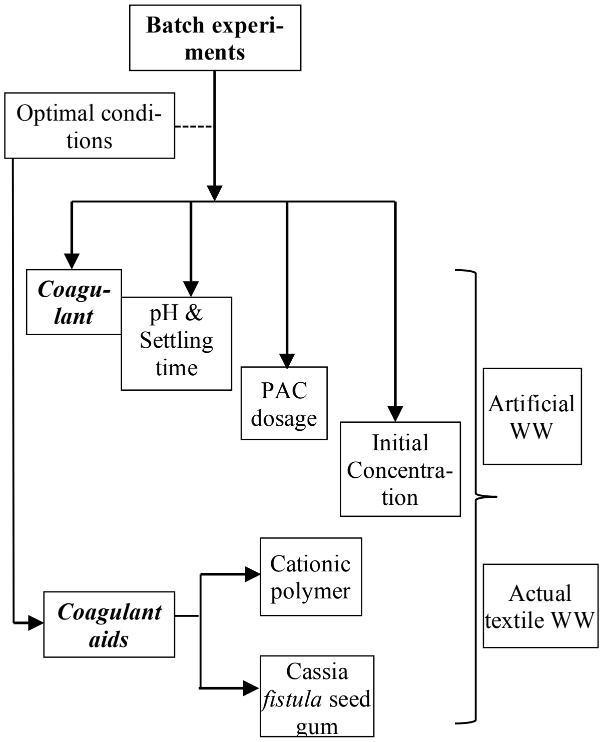

Figure 3. The studying scheme

2.2.1. Batch reactor set up

A Lovibond Jar-test was utilized in batch experiments to determine the optimal conditions of the coagulation_flocculation process with the artificial wastewater, including:
- The sample containing: 6 places for 6 beakers 0.5 L.
- The mixing vane: 6 impellers that can control the mixing rate and reaction time.

The optimal value of each parameter was examined by experiments in which target parameter fluctuated and the others were controlled; the highest of color removing efficiency indicated for the optimal condition as shown in table 1 and figure 4.

For evaluation of treatment's effectiveness (H%), the following equation was applied:

$$H = \frac{C_0 - C_a}{C_0} \times 100, \%$$

Where:
C_0: The initial concentration of COD (mg/L) or the color (Pt-Co);
C_a: The concentration of COD (mg/L) or the color (Pt-Co) after treatment.

The pH and sedimentation time were observed by experiments at these conditions: pH was changed in the range from 3 to 12 by adding HCl 1M and NaOH 0.1 M, the fast stirring rate was 200 rpm for 1 minute and the slow stirring rate was 45 rpm for 15 minutes, the sedimentation time was 15, 30 , and 45 minutes.

The optimal dosage for PAC in treatment of RB21 and MB was examined at these conditions: the volume of PAC was 0.1, 0.15, 0.2, 0.25, 0.3, 0.35 ml; the pH, sedimentation time obtained from previous experiments; the fast stirring rate was 200 rpm for 1 minute and the slow stirring rate was 45 rpm for 15 minutes.

Table 1. Detail of optimal conditions determination

Parameters	Value
pH	3; 4; 5; 6; 7; 8; 9; 10; 11
Concentration of dye, mg/L	150; 200; 250; 300
The dosage of coagulant and coagulant aids, mg/L	
PAC, ml	0.1; 0.15; 0.2; 0.25; 0.3; 0.35
Polymer (0,05%), ml	0.05; 0.1; 0.15; 0.2; 0.25; 0.3
CFG (0,05%), ml	0.05; 0.1; 0.15; 0.2; 0.25; 0.3
Reaction time, minute	15; 30; 45; 60

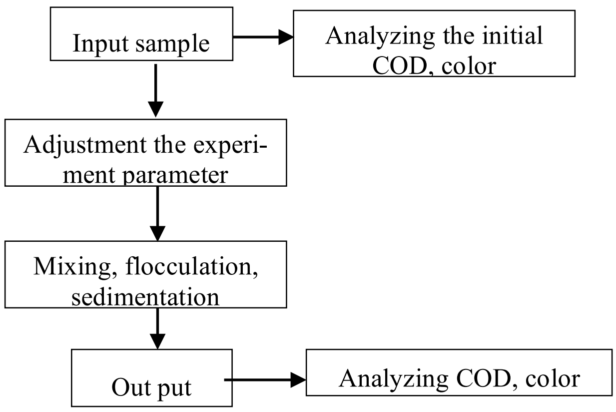

Figure 4. The batch experiment for determination optimal condition.

The best combination of cationic polymer and PAC then determined with the previous optimal parameters and volume of polymer adding was 0.05, 0.1, 0.15, 0.2, 0.25, 0.3 respectively. The best dosage for utilizing CFG was detected by color and COD removing at the volume of CFG 0.05, 0.1, 0.15, 0.2, 0.25, 0.3 mL and other optimal parameters.

After carried out batch experiments with artificial wastewater containing RB21 and MB, actual textile wastewater were treated for evaluating the real ability in COD and color removing under the optimal conditions. Parameters were evaluated included: pH, PAC dosage, CFG aid dosage, and cationic polymer aid dosage.

2.2.2. Analyzing methods

Samples were analyzed for such parameters as pH, COD, and color; following the standard methods. UV-VIS spectrophotometry was used to determine the absorb capacity in COD analyzing. The color of the sample was detected according to Platinum-Cobalt color standard test method for liquid (APHA, 1999).

3. Results

3.1. Effect of pH and sedimentation time

It has been improved in various research that pH plays an important role in the coagulation_flocculation process because it not only affects on the characteristic of reagents but also influents the hydrolysis and solubility of dye. The result of color removal by PCA depending on pH has shown in figure 6. The efficiency in color treatment has changed as the pH changed and the optimal of pH was different due to the kind of dye. Besides, the time of sedimentation has a significant effect on the final outcomes. In particularly, at pH = 4, and sedimentation time = 30 minutes, the color removing yield reached to 93%. In the case of Methyl Blue, pH = 6 has the most impact, with the settling time of 30 minutes, almost 50% of MB was reduced. It could be seen that at lower pH the efficiency was higher that was similar to the finding of Muhammad Asif Hanif (2008). In the real performance of colored wastewater, the hydrolysis of dye has fluctuated by the pH. At the alkaline pH, its hydrolysis is faster than the coagulation process, so that dye can not bind to the PAC. At acidic and neutral pH, the hydrolysis becomes slower and improves the combination of dye material and PAC (Perng, Yuan- Shing, 2014).

It was reported by Peavy et al. that pH influenced to the coagulation at different ranges varying by the dye and coagulant characteristics. If pH is below the isoelectric point of metal hydroxide while precipitation enhanced by a suitable polymer, the positively charged polymers will prevail and predominate the negatively charged colloids by charge neutralization. Above the isoelectric point, anionic polymers will predominate so that particle is adsorbed and develop the bridge. At high dose of metal ion coagulants, a sufficient degree of oversaturation occurs to produce a rapid precipitation of a large quantity of metal hydroxide, catching the colloidal particles to build floc (Peavy et al., 1985).

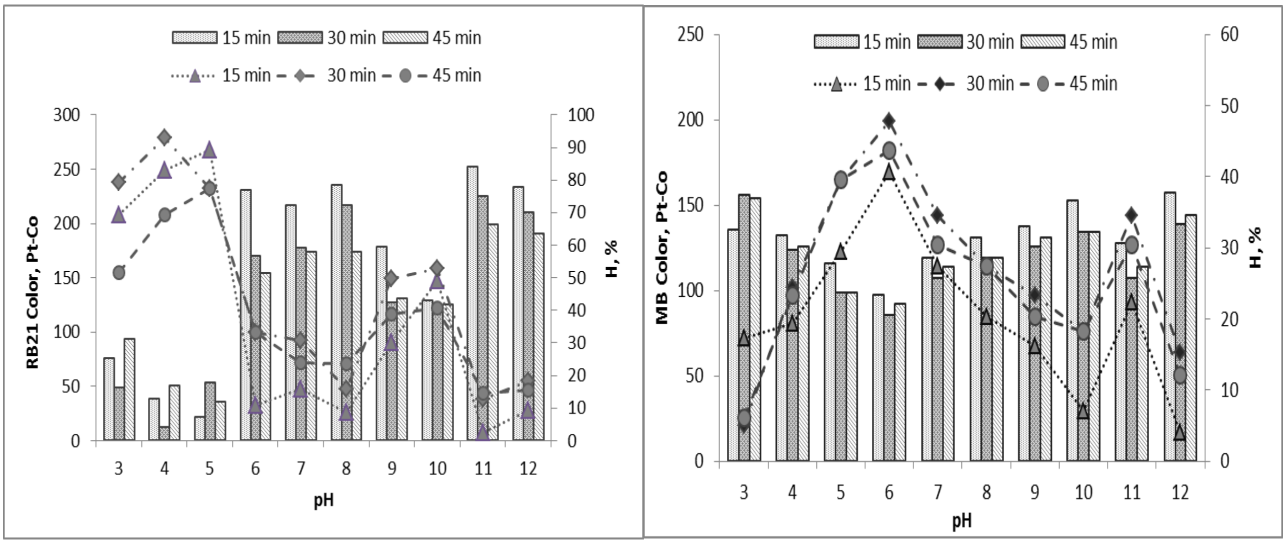

Figure 6. The effluent's color and efficiency of removing of RB21 (a) and MB (b).

3.2. Effect of PAC dosage

Coagulant dose has considered a most important indicator for coagulation_flocculation. As pointed in figure 7, the increasing of PAC dose from 0.1 mL to 0.2 mL has enhanced the proportion of color and COD treatment. Adding PAC to the system more than 0.2 mL has restricted the process. At lower concentration of PAC, the positive charges are not sufficient to destabilize the dye particles than that at higher PAC dosage. The effectiveness in the treatment of COD and color increased by the supplementation of coagulant but at overdosage value, the PAC potential will be lowered. This phenomenon was explained by the fact that the colloid would be re-stabilized at high concentration of coagulant (Metcalf & Eddy, 1991; Le Hoang Viet, Nguyen Vo Chau Ngan, 2014).

At optimal conditions of pH and PAC dosage, the removing percentage of color was around 90% for RB21 and nearly 60% for MB. A similar result was found by Perng, Yuan- Shing and Manh Ha Bui (2014) when they used PAC to treat the colored dyeing wastewater.

3.3. The efficiency of cationic polymer

Furthermore, PAC at low concentration would diminish the coagulation even at optimal pH due to the fact that there was not enough positive charge to make the colloid unstable before they can aggregate into a larger particle. The auxiliary coagulant has used for enhancing the flocculation of suspended particular is wastewater (figure 8). In the assistance of cationic polymer at 0.1 for MB and 0.2 for RB21, the COD removing's efficiency was increased to 96% and 80%, respectively. The color after treatment was also reduced significantly at those conditions. The other studies have indicated that the cationic polymer has a strong effect to the wastewater that has contained a high value of organic matter. In these reactions, cationic polymer has a high ability to attract the suspended solid and neutralize their surface charge so that it helps to reduce the PAC using, produce a small volume of more concentrated and rapid settled floc.

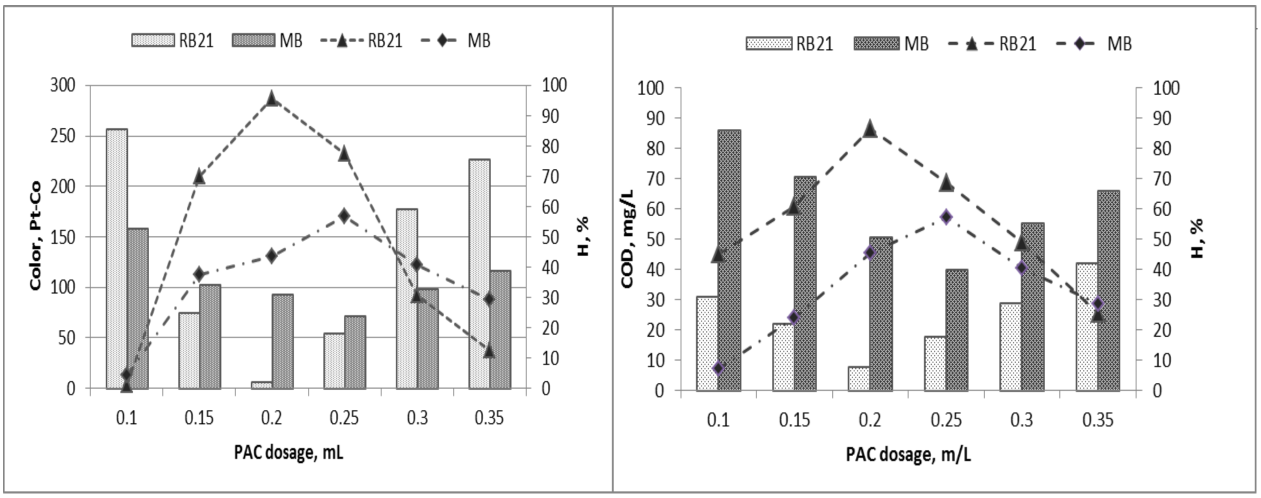

Figure 7. The efficiency of removing color (a) and COD (b) at different PAC dose.

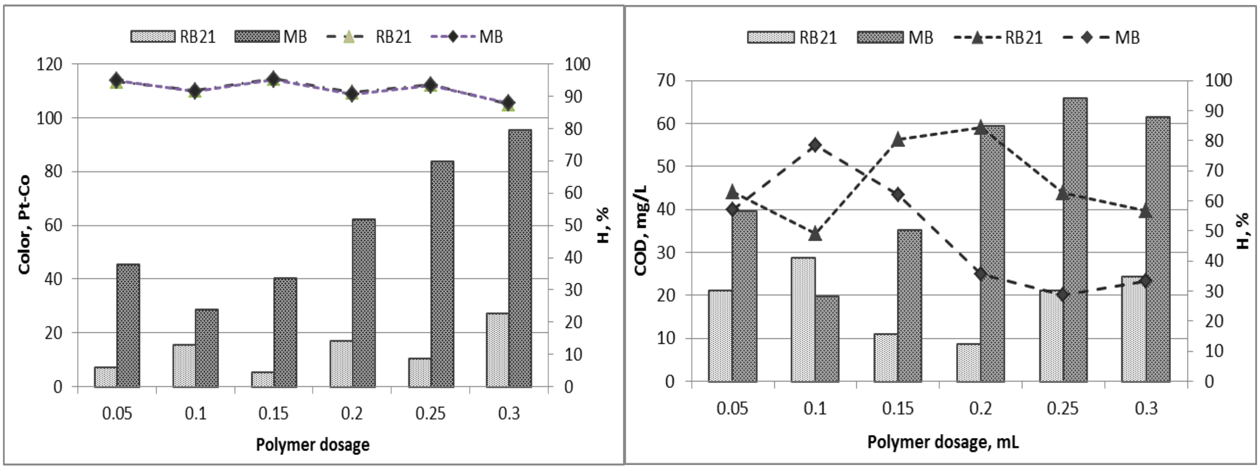

Figure 8. The impact on color and COD removal of polymer dosage.

However, the redundant of the polymer can contribute to COD consequences in the lowering of effectiveness. The polymer that cannot contact to another charged particle will be bent and automatic contact to another surface of itself. So that, the high density of polymer has made the surface' colloids became saturated, they do not have enough space for binding, In this case, the colloids will be re-stabilized indicating for lower effect.

3.4. Effect of Cassia *fistula* seed gum

Adding CFG at the concentration of 0.05% could removal color and COD significantly. The performance of CFG at various dose was represented in figure 9. Obtaining indicated that CFG assists the flocculation process with treatment nearly 98% color for RB21 and 90% color for MB at 0.15 mL and 0.1 mL, respectively. In the treatment of COD, the yield was quite considerable, at about 85% for RB21 and 70% for MB at the concentration of 0.05% can removal color and COD significantly. The endosperm's CFG contain an amount of the galactomannan polysaccharides including the cis-hydroxyl group which can interact with colloidal particles.These findings demonstrated for the suitable of CFG as an auxiliary in color and COD removing in comparing with the chemical polymer. With the similar performance, biodegradable, less pH dependence, and the easy to manufacture CFG can become a promising reagent for green treatment of wastewater.

3.5. The effect of CFG and cationic polymer in color removing of textile wastewater

In order to apply achievements with synthetic wastewater to the real operation, it is important to conduct the experiments with actual textile wastewater obtaining from the actual company in Binh Duong province. Figure 10 shows the effect of pH on color removal and figure 11 indicates the influence of agents' dosage to color reducing of textile wastewater.

The optimized parameters for the coagulation of textile wastewater using PAC were pH = 6 and dose = 0.6 mL could removal 66% of color. By adding CFG to PAC, the efficient of treatment was increased about 70% even at the lower dosage of PAC and CFG (0.5 mL for each reagent). This trend could be explained by the combination of CF gum and PAC providing for a strong chelation of PAC–gum–dye or gum–PAC –dye that attached dye particles to form heavier sludge's flocs and sequenced in the ease to settle (R. S. Blackburn, 2004). The yield of combining PAC and polymer was a little bit lower than PAC and CFG, for instant 68% color was decreased at the same condition.

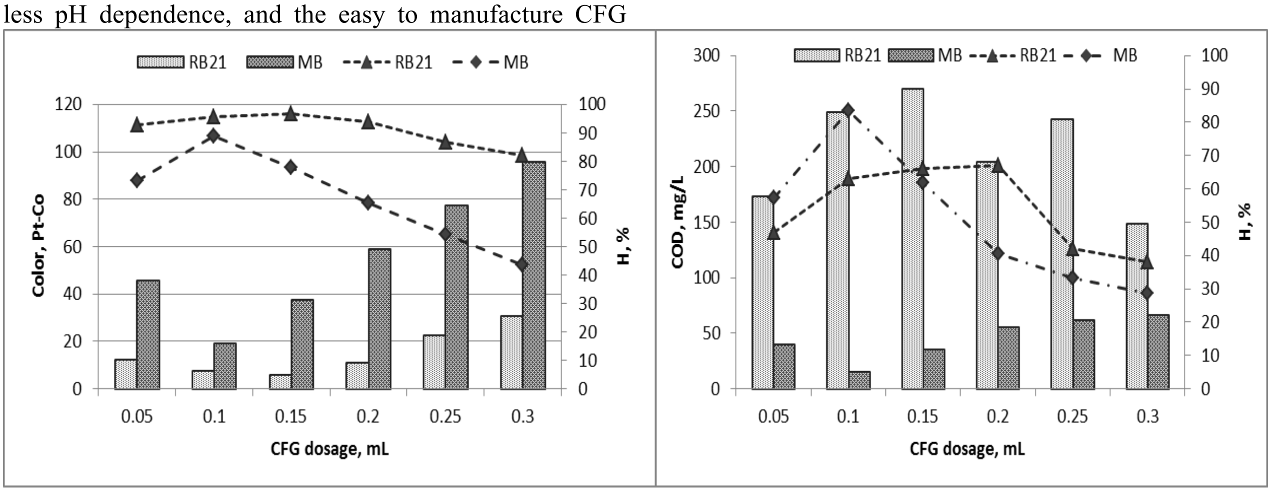

Figure 9. The impact on color and COD removal of CFG dosage

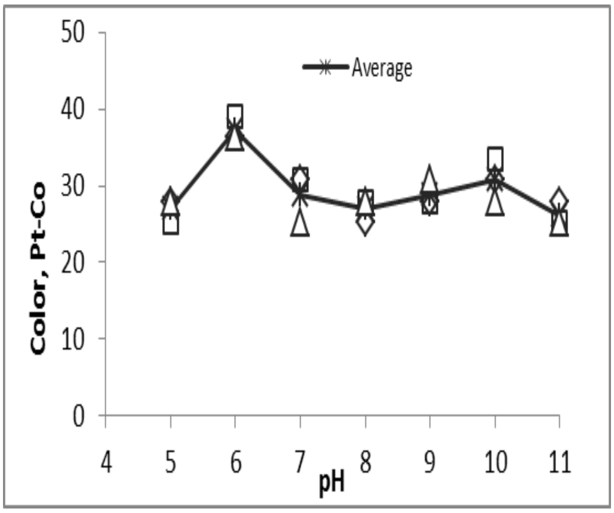

Figure 10. Effect of pH on color removal of textile wastewater.

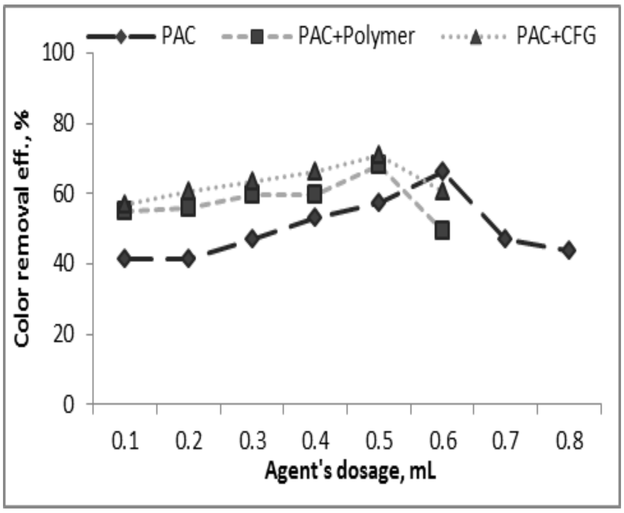

Figure 11. Effect of agent's dosage on color removal of textile wastewater.

4. Conclusion

Obtains from the batch mode treatments of synthetic wastewater containing RB21 and MB indicated that the optimal condition for color and COD removal by using PAC were: pH = 4 for RB21 and pH = 6 for MB; the settling time was 30 minutes, the PAC dosage was 0.2 mL. At optimal conditions of pH and PAC dose, the removing percentage of color was around 90% for RB21 and nearly 60% for MB.

The supplement of the cationic polymer with PAC at 0.15 mL for RB21 and 0.1 mL for MB increased the color removal efficiency to 96% and 80%, respectively. CFG assists the flocculation process at nearly 98% color for RB21 and 90% color for MB at 0.15 mL and 0.1 mL, respectively. In the treatment of COD, the yield was quite considerable, at about 85% for RB21 and 70% for MB at the concentration of 0.05% can removal color and COD significantly.

The optimized parameters for the coagulation of textile wastewater using PAC were pH = 6 and dosage = 0.6 mL can removal 66% of color. In addition of CFG to PAC, the efficient of treatment was increased about 70% even at the lower dosage of PAC and CFG (0.5 mL for each reagent). The yield of combining PAC and polymer was a little bit lower than PAC and CFG, for instant 68% color was decreased at the same condition.

It could be concluded from above findings that CFG natural coagulant aid created a significant impact on the physical treatment of textile wastewater. Besides, with the eco-friendly characteristic and a low-cost method, CFG should be produced and applied in coagulation-flocculation treatment various kind wastewater. We recommend conducting more experiments at the pilot scale in order to evaluate the feasibility of CFG as a coagulant with PAC for the treatment of continuous textile wastewater based on results of this research.

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